PB81-175911

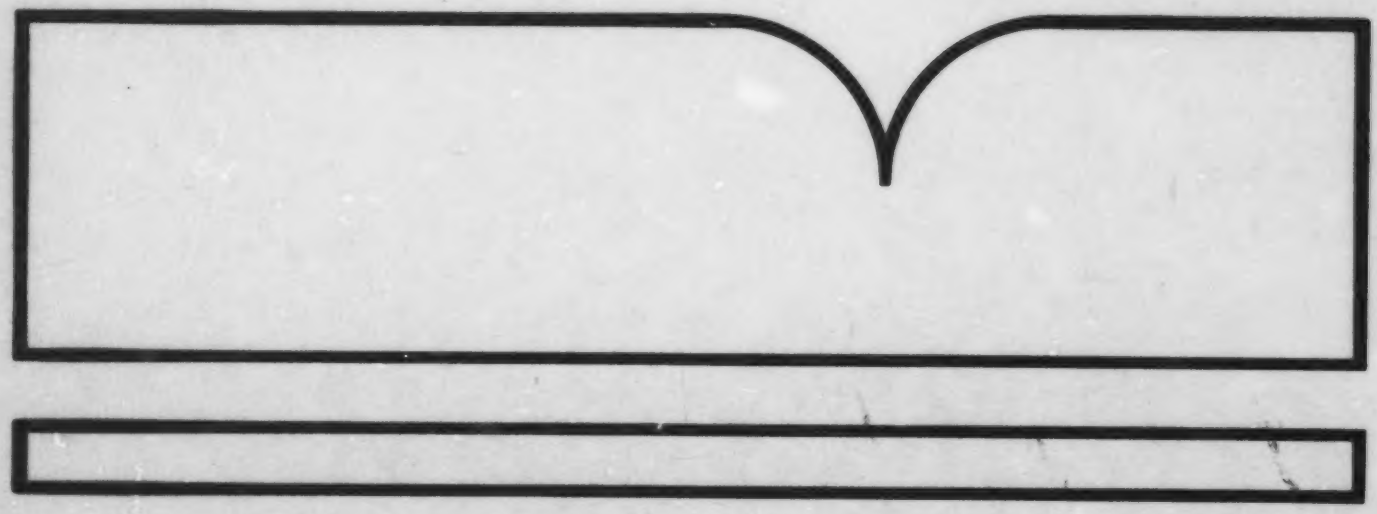
Evaluation of Sampling Techniques for Atmospheric Emissions from Sintering in the Iron and Steel Industry

PEDCO-Environmental, Inc.
Cincinnati, OH

Prepared for

Environmental Monitoring Systems Lab.
Research Triangle Park, NC

Apr 81



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TECHNICAL REPORT DATA
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1. REPORT NO.	2.	3. RECIPIENT'S ACCESSION NO.
EPA-600/4-81-023		PB81 175911

4. TITLE AND SUBTITLE	5. REPORT DATE
EVALUATION OF SAMPLING TECHNIQUES FOR ATMOSPHERIC EMISSIONS FROM SINTERING IN THE IRON AND STEEL INDUSTRY	April 1981
	6. PERFORMING ORGANIZATION CODE
7. AUTHOR(S)	8. PERFORMING ORGANIZATION REPORT NO.
PEDCo Environmental, Inc.	
9. PERFORMING ORGANIZATION NAME AND ADDRESS PEDCo Environmental, Inc. 11499 Chester Road Cincinnati, OH 45246	10. PROGRAM ELEMENT NO.
	11. CONTRACT/GRANT NO. 68-02-2725
12. SPONSORING AGENCY NAME AND ADDRESS Environmental Monitoring Systems Laboratory Office of Research and Development U.S. Environmental Protection Agency Research Triangle Park, NC 27711	13. TYPE OF REPORT AND PERIOD COVERED Final
	14. SPONSORING AGENCY CODE EPA-600/08

15. SUPPLEMENTARY NOTES

To be published as an ORD Project Report

16. ABSTRACT

Tests were conducted at two sintering plants of the Iron and Steel Industry to evaluate a test method under consideration by the U.S. Environmental Protection Agency. In field tests, four modified Method 5 sampling trains, operating simultaneously at a single point in the stack, yielded four samples per sampling run. Each sample was analyzed for front-half filterable particulates and back-half organic solvent extractables (OSE). In addition, the effects of filter and probe temperatures on filter collection efficiency, and the effects of sample handling, storage conditions, and recovery procedures on sampling results were studied. Temperature fluctuations from 65° - 120°C (150° - 250°F) did not significantly affect the particulate concentrations, nor did additional dessication greater than the conventional time period. Chromatographic experiments indicated that pH 7 was preferable for OSE extraction.

17. KEY WORDS AND DOCUMENT ANALYSIS

a. DESCRIPTORS	b. IDENTIFIERS/OPEN ENDED TERMS	c. COSATI Field/Group
Sampling Techniques Sintering		43F 68A

18. DISTRIBUTION STATEMENT	19. SECURITY CLASS *(This Report)*	21. NO. OF PAGES
RELEASE TO PUBLIC	Unclassified	
	20. SECURITY CLASS *(This page)* Unclassified	22. PRICE

EPA Form 2220-1 (Rev. 4-77) PREVIOUS EDITION IS OBSOLETE

PB81-175911

EPA 600/4-81-023
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EVALUATION OF SAMPLING TECHNIQUES FOR ATMOSPHERIC EMISSIONS FROM SINTERING IN THE IRON AND STEEL INDUSTRY

by

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Contract No. 68-02-3431
Task No. 33
PN 3529-7

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ENVIRONMENTAL MONITORING SYSTEMS LABORATORY
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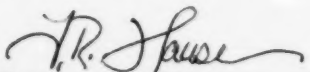
FOREWORD

Measurement and monitoring research efforts are designed to anticipate potential environmental problems, to support regulatory actions by developing an in-depth understanding of the nature and processes that impact health and the ecology, to provide innovative means of monitoring compliance with regulations and to evaluate the means of monitoring compliance with regulations and to evaluate the effectiveness of health and environmental protection efforts through the monitoring of long-term trends. The Environmental Monitoring Systems Laboratory, Research Triangle Park, North Carolina, has responsibility for: assessment of environmental monitoring technology and systems; implementation of agency-wide quality assurance programs for air pollution measurement systems; and supplying technical support to other groups in the Agency including the Office of Air, Noise and Radiation, the Office of Toxic Substances and the Office of Enforcement.

The following investigation was conducted at the request of the Office of Air Quality Planning and Standards. Test methods for the measurement of particulate emissions were evaluated. The work included studies of techniques and procedures for the measurement of both filterable and condensible particulate using the conventional EPA reference method 5 at sintering plants in the Iron and Steel Industry.



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Tests were conducted at two sintering plants of the Iron and Steel Industry to evaluate a test method under consideration by the U.S. Environmental Protection Agency. In field tests, four modified Method 5 sampling trains, operating simultaneously at a single point in the stack, yielded four samples per sampling run. Each sample was analyzed for front-half filterable particulates and back-half organic solvent extractables (OSE). In addition, the effects of filter and probe temperatures on filter collection efficiency, and the effects of sample handling, storage conditions, and recovery procedures on sampling results were studied. Temperature fluctuations from 65° - 120°C (150° - 250°F) did not significantly affect the particulate concentrations, nor did additional dessication greater than the conventional time period. Cromatographic experiments indicated that pH 7 was preferable for OSE extraction.

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A three volume supplement to this report containing computer printouts, field data sheets, laboratory results, sampling and analytical procedures, calibration procedures and results, and a sample analyses report prepared by Research Triangle Institute are available for review through the Quality Assurance Division of the Environmental Monitoring and Systems Laboratory, MD-77, Research Triangle Park, North Carolina.

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SECTION 1

INTRODUCTION

Sintering is a process used by the Iron and Steel Industry to convert finely divided, iron-bearing materials, such as ore fines and mill scale, into a granular material that can be put into the blast furnace. These iron-bearing materials are mixed with a limestone flux, coal or coke, and water to obtain a composite material with the desired composition and pH. The composite material (burden) is put on a traveling grate. Combustion, initiated by an auxilary fuel, becomes self-supporting, and enough heat is generated, 1315-1480°C (2400-2700°F), to agglomerate the fine iron-bearing materials into a granular material called the sinter cake. At the end of the traveling grate, the sinter cake is broken into pieces of about 10 to 15 cm (4 to 6 in.) by a machine called a sinter breaker. Windboxes are compartments under the traveling grate that enable uniform distribution of combustion air through the sinter bed. This air passes through the sinter bed, into the windbox, and then through a baghouse, a wet scrubber, or an electrostatic precipitator, and into the stack.

The two major points for emissions are the sinter breaker vent and the windbox vent. Emissions from the sinter breaker are generally thought to consist only of filterable particulates--materials collectable by a standard Method 5 glass fiber

filter maintained at approximately 120°C (250°F). However, condensible materials collectable in the impingers may also be emitted from the sinter breaker vent, the result of emissions from the sinter coke.

Besides filterable particulates, the emissions from the windbox can contain NH_4^+, SO_2, H_2SO_4, NO_x, F, and condensible organic materials that can pass through a heated glass fiber filter, and may condense in the impinger part of the Method 5 train. Some of these condensible materials are soluble in organic solvents, and will be termed organic-solvent extractables (OSE) to indicate that this catch depends on the means of recovery.

Organic materials could come from the coke or the coal added to the iron-bearing material, but it is more likely that the organics come from mill scraps which contain cutting oils. Another potentially significant source of the condensible emissions are not organic at all, but are inorganic compounds such as mineral acids (H_2SO_4, HF, HCI) and inorganic salts (NH_4SO_4, NH_4Cl, $FeCl_3$, $PbCl_2$, KCl and metal oxides). The difference in the amounts of these inorganic materials may account for some of the variation is concentrations observed between emissions from each source and between sampling runs from the same source.

Emission testing was conducted at two sintering plants of the Iron and Steel Industry to evaluate a tentative test method for measuring filterable particulate and filterable plus nonfilterable OSE emitted from the stacks of the plants. In this

tentative method, the sampled stack gas is first passed through a heated probe into a heated filter assembly (the front half) for collecting filterable particulates, and then the gas is drawn through a series of four impingers (the back half). Each of the first two impingers contains 100 ml of distilled water for collecting the condensible particulates, the third is dry for collecting water carryover, and the fourth contains 200 g of silica gel for removing water vapor. (In several method development tests, a nonheated filter was placed between the third and fourth impingers to collect any filterable sample not collected in the rest of the sampling train.) The filterable particulates were measured by the procedure in EPA Reference Method 5.[1] The organics were extracted with an ether-chloroform extraction method.

For both sampling sites, four sampling trains were operated simultaneously at a single point in the stack yielding four samples per run, which could be studied as a block. For both Plant A and Plant B, several pieces of data were eliminated as outliers; these data are described in the Test Design and Results Sections.

It should be noted that Plant A was of a special design having a much larger than usual process feed rate but a smaller concentration of condensible material. Process operating conditions were more uniform and emissions more consistent than at the other plant tested. Plant B, on the other hand, was of a more conventional design handling a variety of scrap iron-bearing materials. The feed rate and operating conditions were more

variable, though, and testing was interrupted on several occasions due to upsets on the sinter process line. Windbox emissions were controlled by a high energy venturi scrubber at both Plants A and B.

SECTION 2

CONCLUSIONS AND RECOMMENDATIONS

SAMPLING-TEMPERATURE EFFECTS

A major problem with Method 5 sampling is the collection of unwanted condensible material on the filter. It had been expected that more front-half material and less OSE back-half material would be collected if the filter temperature were reduced. Data on separate collections at 120°C (250°F) and at 65°C (150°F) could not be compared because the process conditions and feed materials were not constant over the test period of several days, but data on simultaneous run sets, where samples from the first two trains collected at 120°C (250°F) and those from the other two collected at 65°C (150°F) were compared. At Plant A and Plant B, reduction in the filtration temperature from 120°C (250°F) to 65°C (150°F) did not cause the front-half catch to increase significantly. A temperature reduction sufficient to cause a measurable condensation of organics was not possible because condensation of moisture from the gas stream wet the filter, and the vapor pressure of the materials (even at 65°C (150°F)) was enough to prevent such low concentrations of materials from condensing on the filter.

Water soluble sulfate measurements were made to determine if the sample filtration temperature affected the collection of

the sulfates. Similar to the process for condensible organic collection, a lower filtration temperature would be expected to condense more sulfuric acid mist resulting in either a higher filterable sulfate concentrations or a lower impinger sulfate concentrations. Again, the data did not show any significant bias in sulfate concentration due to filtration temperature.

Therefore, it may be concluded that temperature fluctuations from 120°C (250°F) to 65°C (150°F) will not significantly affect the particulate concentrations by increasing the deposits of condensible materials on the filter. It is recommended that the standard EPA reference method 5 filtration temperature of 120°C (250°F) be used.

pH EFFECTS

The amount of OSE collected by a Method 5 train is usually considerably less than 100 mg, but the volume of organic solvents usually exceeds 100 ml. Under these conditions, substantial amounts of metal sulfates, nitrates, and oxides can be dissolved by the organic solvent.[2] The amounts extracted depend mainly on the solvent and on the pH of the solution.[3] The data on Plant A and Plant B show that sequential extractions at acid, neutral and basic pH's cause materials to be collected with each extraction. The OSE concentrations at Plant A were determined with the solution at pH 3. Chromatographic experiments at Plant B indicated that pH 7 was preferable so the tests for OSE were conducted with the solution at that pH.[4] Therefore, it is recommended that the impinger solution be adjusted to pH 7.

ALTERNATIVE CONDENSER SYSTEM

The glass condenser system tested at Plant A proved acceptable as an alternative to the standard impinger arrangement for collecting condensible particulates, although no advantages could be found for using this method.

DRYING-TEMPERATURE EFFECT

Filterable particulate samples exposed to elevated temperatures for fixed time periods did not lose appreciable mass after the initial gravimetric determinations. In this way it was determined that filter drying conditions have no serious effect on sample loss. However, oven drying of the filter was not recommended for the general case since conventional desiccation was satisfactory.

TOTAL ORGANIC CARBON (TOC) VS. EXTRACTION

Analysis of impinger contents by a TOC analyzer yielded unacceptable, irreproducible results when compared to the values resulting from the ether-chloroform extraction method. A cutting-oil study suggested that a homogeneous sample is needed for TOC analysis, but the laboratory recovery of extracts by ether-chloroform is possible with good accuracy and precision even in a nonhomogeneous sample.

HYDROCARBON ANALYSIS BY FLAME IONIZATION DETECTOR (FID)

Analysis of gas leaving the backup filter (between the third and fourth impingers) using a gas chromatograph (GC) FID at Plant A showed that the sampling train removed 98% of the material that gave a response on the GC/FID. (Note: This analysis was not conducted at Plant B.)

ORGANIC MATERIAL REMOVAL USING METHYLENE CHLORIDE

Recovery procedures for the sample in the impinger portion of the sampling train at Plant B consisted of recovering the impinger water, rinsing it with distilled water, rinsing it with acetone, and finally rinsing it with methylene chloride. (Methylene chloride rinses were not included in the procedure at Plant A.) Methylene chloride is an excellent degreaser solvent; it removed approximately five times more material than the standard rinse alone, but it is hazardous for field use, and is not recommended unless extreme caution is taken during handling and sampling. Subsequent organic analysis of the fraction rinsed by methylene chloride found it to contain a single GC peak not typical of materials found at sintering plants. One possible explanation is that the methylene chloride removed the impinger silicone grease which the acetone did not dissolve, yet sufficient data is not available to substantiate this statement.

IMPINGER SULFATE CONTENT

The sulfates formed in the cooled impinger water constitute a large portion of the inorganic materials in the back-half catch, although the sulfates found there may possibly be byproducts of sulfuric acid mist in the sintering process. The inorganics in the back-half catch were variable, and may indeed include pseudo-particulates which would not normally form in ambient air. Because these results were erratic and irreproducible, the inorganic (nonextractable) portion of the back-half catch should not be included as part of the total catch.

GENERAL CONCLUSIONS ON THE USE OF THE EPA REFERENCE METHOD 5 EMPLOYING THE OSE CATCH

No problems were apparent with the field use of EPA Reference Method 5 for testing at the Iron and Steel Industry sintering plants. Precision of the data on the filterable particulate catch was roughly in line with test data using Method 5; the coefficient of variation (CV), which is the standard deviation (SD) divided by the mean, was about 19%. The OSE results by themselves present a different picture; there was much more variation. These studies did not specifically identify what caused the lack of precision, but did demonstrate that for grouped samples taken at the same time from the same location in the stack the CV in the OSE catch varied from 20 to 40%. Based on the data from these two plants, the effect of the OSE data variability was not significant when total catch (filterable particulate plus OSE) was calculated; in fact for some runs the precision of the total catch was better than either the filterable particulate or OSE alone. Therefore, the use of the OSE catch in combination with the filterable particulate catch does not adversely affect the total catch and the total catch can be used to estimate the total particulate emitted.

SECTION 3

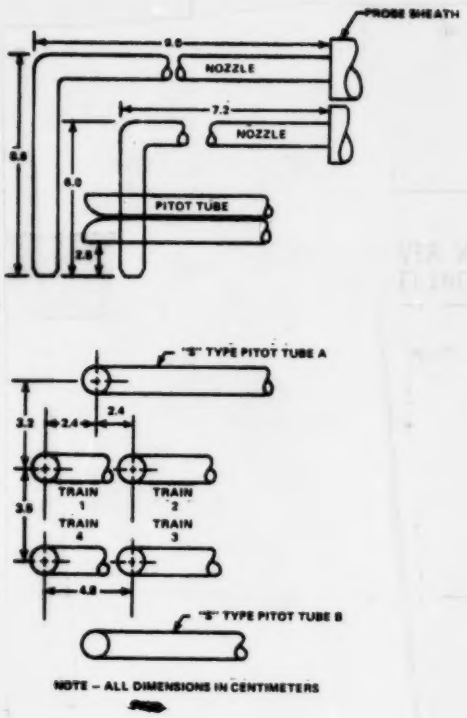
TEST DESIGN

Testing at Plant A was performed at two sites--the windbox and the breaker discharge. The windbox emissions were controlled with a high-energy venturi scrubber while the breaker discharge emissions were controlled with high-efficiency multicyclones. The test was designed to evaluate the collection efficiency and reproducibility of the tentative method. It was planned to improve the method as needed to take maximum advantage of the data as it was acquired. Emissions from Plant B were sampled at the windbox only; this plant is typical of sintering plants that use scrap from other processes as the feed.

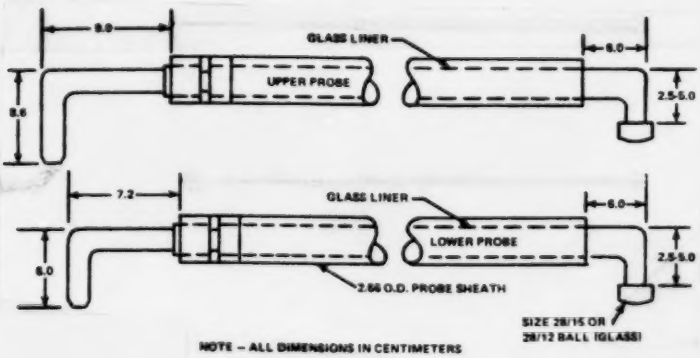
To allow for valid comparisons, a four-train sampling design was used.[5] The sampling arrangement used in the tests consisted of two sets of two sampling trains operating independently of each other, except that both trains in the same set used the same Type S pitot tube for setting and maintaining an isokinetic sampling rate. This system obtained reliable estimates of emission concentrations and permitted the evaluation of the precision of the tentative method and of the alternative methods.

Figures 1 through 6 illustrate the important features of the sampling equipment. Figure 1 shows the orientation of the

sampling probes; Figures 2 and 3 show how two sets of trains are assembled for easy entry into and removal from the stack; Figure 4 shows the filter box heater designed to provide constant heating of the filters. Figures 5 and 6 show a glass condenser system designed and fabricated by EPA for collecting condensible particulates. This system was also evaluated through field and analytical studies by Research Triangle Institute laboratories.[4]

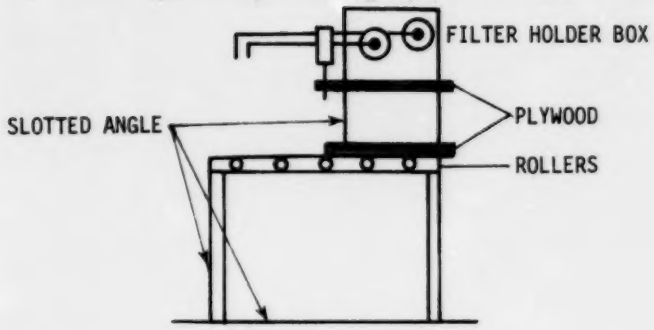


Double Pitot Sampling Arrangement: (Top) Side View; (Bottom) Upstream View.



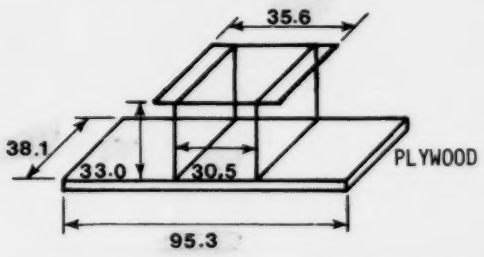
Upper and Lower Sampling Probes.

Figure 1. Double pitot sampling arrangement.

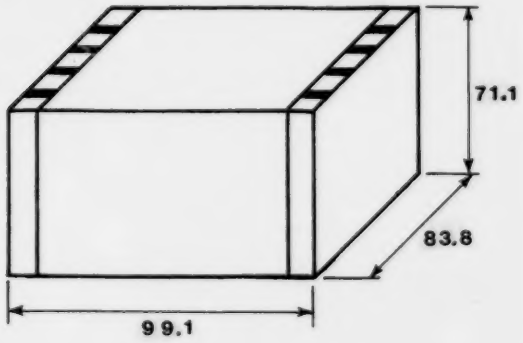


SIDE VIEW

EXPANDED VIEW, ROLLING ASSEMBLY



EXPANDED VIEW OF SLOTTED ANGLE BASE



NOTE - ALL DIMENSIONS IN CENTIMETERS

Figure 2. Train stand assembly.

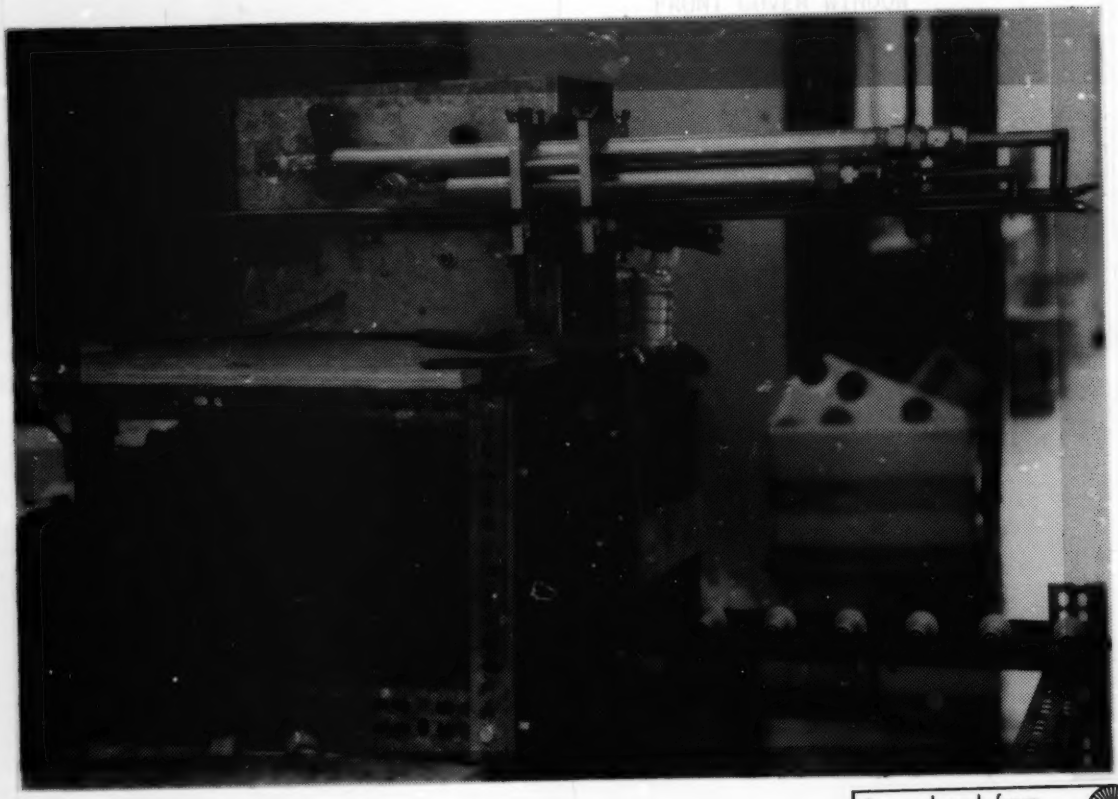


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Figure 3. Pitot tube and probe assembly.

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Figure 4. Heated filter box.

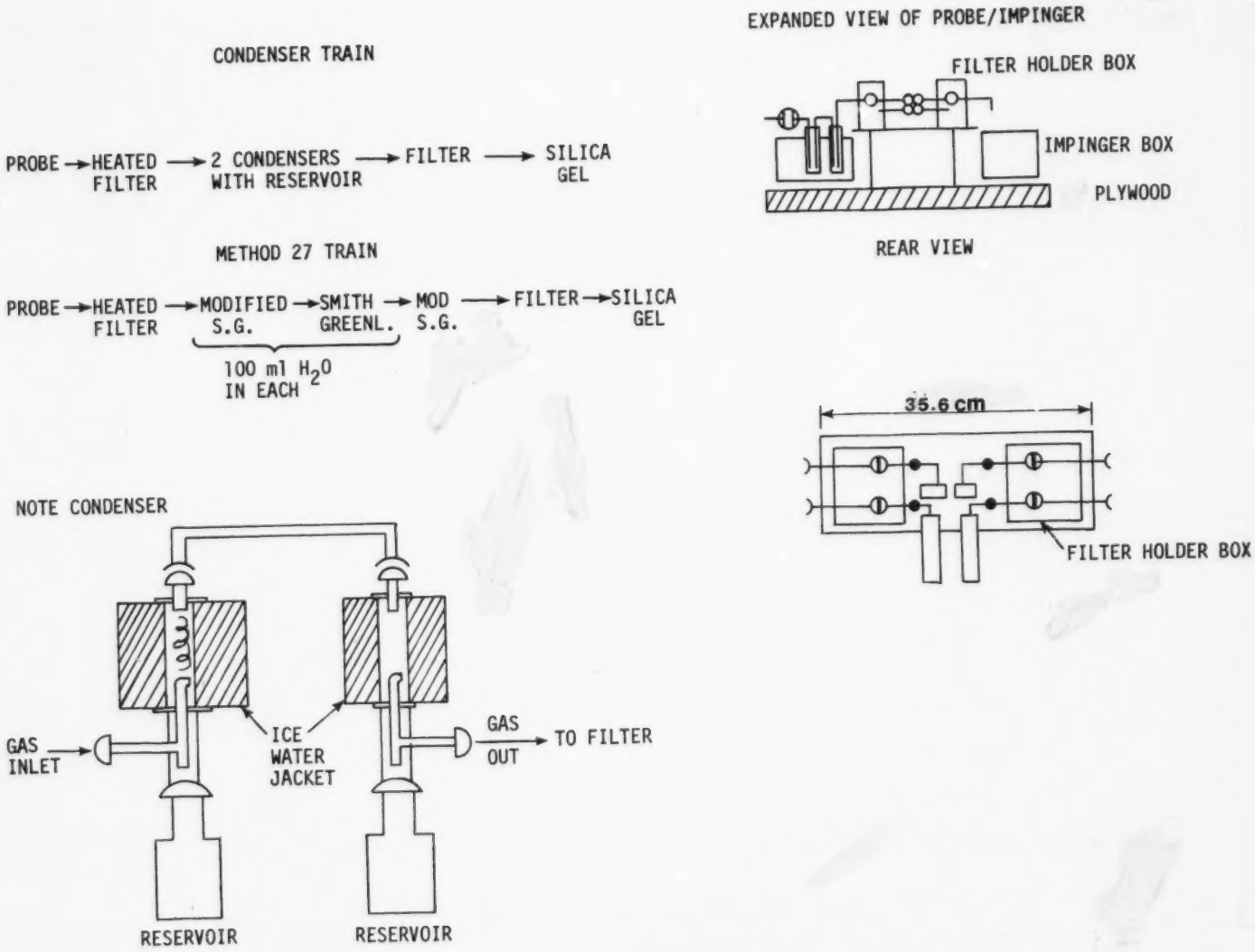

Figure 5. Condenser train assembly.

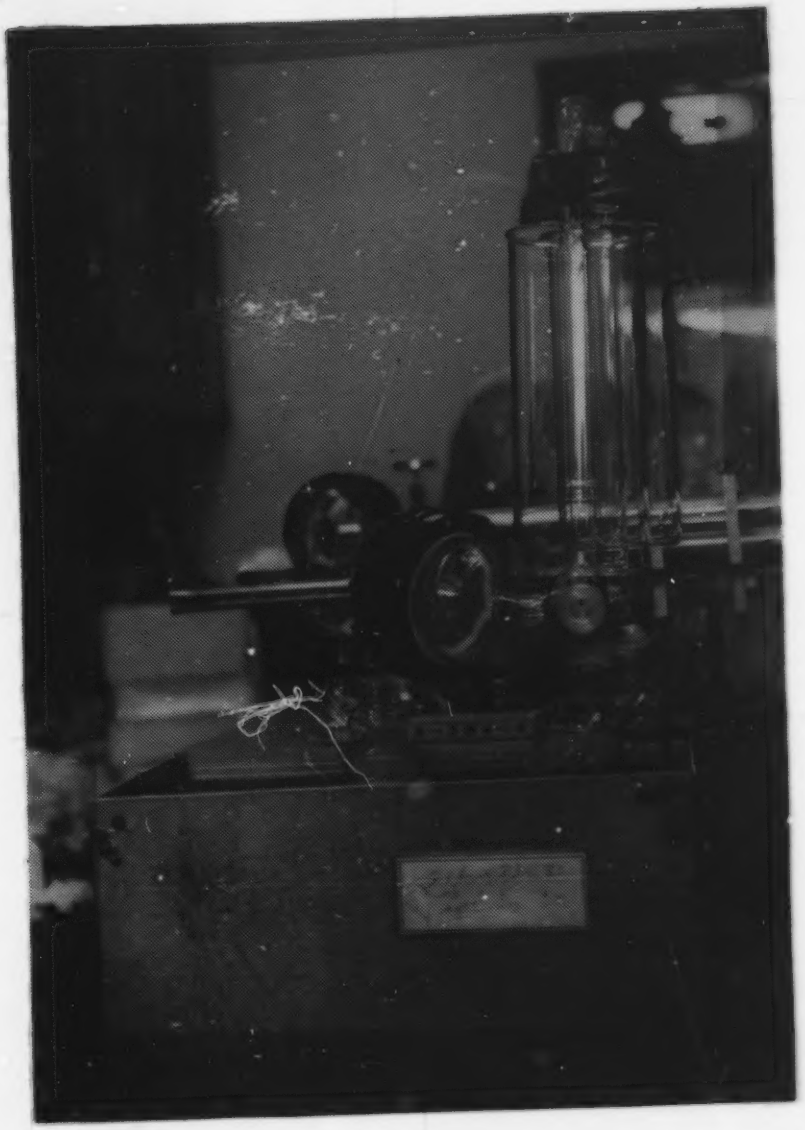

Figure 6. Condenser train.

SECTION 4

RESULTS

RESULTS OF PLANT A

The results of the analysis of the emissions from the windbox and breaker discharge of Plant A are presented in Table 1. The breaker discharge results will not be discussed in the text. Table 2 presents the statistical analysis of the grouped run sets data.

The concentrations of filterable particulates averaged 28.7 milligrams per standard cubic meter (mg/sm^3) with a SD of 3.4 mg/sm^3 for the 25 windbox samples collected with a filter temperature of 120°C (250°F). At the same temperature, without the condenser system, the OSE concentrations collected in the impingers averaged 4.5 mg/sm^3 with a SD of 2.03 mg/sm^3. The average concentration for the total particulates (front-half catch and OSE) was 33.4 mg/sm^3 with a SD of 4.2 mg/sm^3 and for impinger inorganics, 38.6 mg/sm^3 with a SD of 15.1 mg/sm^3. The front-half catch accounted for 85.9% of the total particulates and 40.6% of the entire train contents (front-half, OSE, and inorganics) at Plant A.

Data Precision

The precision of the data on filterable particulates was estimated by calculating the CV. The average CV for all valid

TABLE 1. RESULTS FROM PLANT A[a]

Test number	Sample[b] volume	Emission concentrations, mg/sm³				
		Filterable particulate	Filterable[c] sulfate	Impinger pH-3 OSE	Total[d]	Impinger inorganics
Windbox--filter at ≅ 120°C (250°F)						
1-A	1.36	34.54	-	9.46	44.00	26.91
1-B	1.55	34.50	-	5.04	39.54	21.67
1-C	1.62	28.71	-	3.64	32.35	31.11
1-D	-	-	-	-	-	-
2-A	2.35	27.80	-	3.41	31.21	22.47
2-B	2.43	28.30	-	3.41	31.71	21.07
3-A	2.61	28.01	2.84	4.06	32.07	44.64
3-B	2.63	30.00	6.62	4.33	34.33	65.02
3-C	2.61	32.34	2.87	-	-	56.69
3-D	2.53	30.56	4.66	2.65	33.21	55.57
4-A	2.39	26.97	2.85	5.72	32.69	58.54
4-B	2.42	23.29	4.13	4.59	27.88	36.24
5-A	2.06	27.24	11.70	2.96	30.20	31.31
5-B	2.18	24.84	27.43	-	-	38.07
5-C	2.12	33.70	13.30	5.57	39.27	-
5-D	2.04	31.82	10.93	3.68	35.40	13.92
6-A	2.03	26.25	3.25	-	-	-
6-B	2.08	22.16	3.75	4.40	26.56	-
6-C	2.06	29.86	2.18	3.93	33.79	-
6-D	2.01	31.61	5.72	3.14	34.75	-
7-A	3.14	26.02	3.54	4.01	30.03	39.19
7-B	3.20	23.76	5.59	9.72	33.48	35.18
8-A	1.88	28.13	21.56	4.57	32.70	64.57
8-B	1.90	26.08	20.42	1.52	27.60	34.58
8-C	1.89	31.17	11.80	6.97	38.14	46.61
8-D	1.83	30.30	14.75	2.30	32.60	27.76
Windbox--filter at ≅ 65°C (150°F)						
2-C	2.46	21.10	-	3.00	24.10	18.54
2-D	2.31	18.55	-	2.30	20.85	21.65
4-C	2.49	11.80	1.65	5.50	17.30	39.68
4-D	2.33	19.26	2.23	4.04	23.30	57.21
7-C	3.12	33.55	3.97	4.29	37.84	31.21
7-D	3.04	20.80	4.52	2.83	23.63	35.66

(continued)

Table 1 (continued)

Test number	Sample[b] volume	Emission concentrations, mg/sm^3				
		Filterable particulate	Filterable[c] sulfate	Impinger pH-3 OSE	Total[d]	Impinger inorganics
Breaker discharge--filter at ≅ 120°C (250°F)						
1-B	2.06	39.09	0.39	7.28	46.37	3.98
2-A	1.80	27.42	0.22	1.33	28.75	1.50
Breaker discharge--filter at ≅ 65°C (150°F)						
1-A	1.95	34.81	0.31	2.20	37.01	2.12
2-B	1.98	26.60	0.20	1.21	27.81	2.78

[a]Stack conditions - 55°C (130°F), 20% H_2O.

[b]Dry standard cubic meters.

[c]Water soluble sulfate content of the filterable particulate.

[d]Filterable particulate plus impinger OSE.

data was 12%. In addition to calculating gross averages of the total runs, values within run sets could be compared because the data were collected in simultaneous sets of four samples; the average CV for the grouped data (Table 2) was 13%. This level of precision is about twice as high as that in similar grouped tests at well-controlled power plants and municipal incinerators. However, the data do show that temperature changes of process conditions that existed during the testing did not account for the variation in the concentration of filterable particulates.

For all valid runs, the average CV for the OSE data was approximately 45%; for the data grouped by run sets, the average CV was 30%. The concentrations of OSE were small, and the variations were probably due as much to recovery and analytical technique as to changes in organic content in the process gas stream. A change of approximately 1 mg would account for the difference in the 45% and 30% CV values.

Filter-Probe Temperature Study

The temperatures of the probe and filter were varied to determine the effect of temperature on the filterable particulate catch as well as on the filterable-to-extractable ratio. For the three runs at the reduced filter-probe temperature, the filterable particulate concentrations were approximately 18% lower than those for the corresponding 120°C (250°F) trains.

TABLE 2. STATISTICAL RESULTS OF GROUPED RUN SET DATA
PLANT A

Run set[a]	Mean ($\bar{x}$)			Standard deviation (SD)			Coefficient of variation (CV = SD/$\bar{x}$ × 100)		
	FP[b] mg/sm³	OSE mg/sm³	Total mg/sm³	FP mg/sm³	OSE mg/sm³	Total mg/sm³	FP, %	OSE, %	Total %
1 A-C	32.58	6.05	38.63	3.35	3.04	5.88	10	50	15
2 A, B C, D	28.05	3.41	31.46	0.35	0.0	0.35	1	0.0	1
(150)	19.83	2.65	22.48	1.80	0.49	2.30	9	18	10
3 A-D[c]	30.23	3.68	33.20	1.78	0.90	1.13	6	24	3
4 A, B C, D	25.13	5.16	30.29	2.60	0.80	3.40	10	16	11
(150)	15.53	4.77	20.30	5.28	1.03	4.24	34	22	21
5 A-D[d]	29.40	4.07	34.96	4.07	1.35	4.55	14	33	13
6 A-D[e]	27.47	3.82	31.70	4.18	0.64	4.48	15	17	14
7 A, B C, D	24.89	6.87	31.76	1.60	4.04	2.44	6	59	8
(150)	27.18	3.55	30.74	9.02	1.03	10.05	33	28	33
8 A-D	28.92	3.84	32.76	2.28	2.46	4.31	8	64	13

[a]120°C (250°F) unless otherwise indicated by (150) for 65°C (150°F).

[b]Filterable particulates.

[c]Run C OSE invalid.

[d]Run B OSE invalid.

[e]Run A OSE invalid.

pH Effects

The impinger contents were extracted sequentially at pH 3, pH 11, and pH 7 at Plant A to determine the effects on the total particulate catch. Extractions at pH 11 and pH 7 yielded <10% of that obtained at pH 3, so extraction was done with the solution at pH 3 at this plant.

Condenser-Impinger Study

A glass condenser system was evaluated at Plant A as an alternative to the standard impinger arrangement for collecting condensible particulates. The condenser system could be another method of collecting the condensible fraction of the sample, as shown by Runs 5 and 8 at Plant A (Table 3, note a) but, as it showed no advantages, the system was not studied further.

Time-Temperature Effects

Weight loss in the sample at various times and temperatures was analyzed. The sample was initially dried in a desiccator for 24 h. After the standard gravimetric determinations of the filterable particulates were made, the acetone rinses and the filters were dried an additional 72 h in the desiccator and then weighed. Then they were heated to 200°C (390°F) and reweighed. In almost all cases, the loss was <5%; in many cases, the loss was <1%.

Total Organic Carbon (TOC) Analyzer vs. Ether-Chloroform Extraction

The organic content of the impinger solution at Plant A was measured by two analytical techniques in order to identify a suitable alternate method since the current method requires chloroform, a suspected carcinogen, and ethyl ether, a potentially hazardous and explosive vapor. An aliquot of each sample from the impingers was analyzed by the TOC analyzer, and the remaining sample was analyzed by ether-chloroform extraction. The TOC method was unacceptable since the results from the TOC method were irreproducible and widely variable as compared to

TABLE 3. TOC VS ETHER-CHLOROFORM EXTRACTION, PLANT A

Test number	Sample volume[b]	Emission concentrations, mg/sm³	
		Total organic carbon measured by TOC	OSE measured by ether-chloroform extraction
Windbox--filter at ≅ 120°C (250°F)			
1-A	1.36	6.53	9.46
1-B	1.55	6.14	5.04
1-C	1.62	6.05	3.64
1-D			
2-A	2.35	4.39	3.41
2-B	2.43	5.63	3.41
3-A	2.61	5.59	4.06
3-B	2.63	6.08	4.33
3-C	2.61		
3-D	2.53	8.55	2.65
4-A	2.39	4.26	5.72
4-B[a]	2.42	3.90	4.59
5-A[a]	2.06	5.88	2.96
5-B	2.18		
5-C	2.12	6.09	5.57
5-D[a]	2.04	4.03	3.68
6-A	2.03	7.63	
6-B	2.08	12.77	4.40
6-C	2.06	11.95	3.93
6-D	2.01	8.03	3.14
7-A	3.14	9.19	4.01
7-B	3.20	5.94	9.72
8-A[a]	1.88	11.22	4.57
8-B	1.90	477.99[c]	1.52
8-C	1.89	12.63	6.97
8-D[a]	1.83	259.76[c]	2.30
Windbox--filter at ≅ 65°C (150°F)			
2-C	2.46	3.81	3.00
2-D	2.31	3.86	2.30
4-C	2.49	6.74	5.50
4-D	2.33	7.61	4.04
7-C	3.12	7.14	4.29
7-D	3.04	6.47	2.83

(continued)

Table 3 (continued)

Test number	Sample volume[b]	Emission concentrations, mg/sm³	
		Total organic carbon measured by TOC	OSE measured by ether-chloroform extraction
Breaker discharge--filter at ≅ 120°C (250°F)			
1-B	2.06	4.42	7.28
2-A	1.80	2.16	1.33
Breaker discharge--filter at ≅ 65°C (150°F)			
1-A	1.95	26.91	2.20
2-B	1.98	1.92	1.21

[a]Condenser train.

[b]Dry standard cubic meters.

[c]Data suggests that these values should be considered outliers and therefore, eliminated from further analysis.

those from the ether-chloroform extraction methods (Table 3). The poor showing of the TOC method may have been because the aliquots were not homogeneous or because traces of acetone from the final acetone rinse remained.

Cutting Oil Study

An experiment was developed to explain the differences between the results of the two analytical methods, mentioned previously, which were used to determine the organic content of the impinger sample fraction. Since it was suspected that the samples were an oil, and not homogeneous, known quantities of a commercial cutting oil were used as the organic material for comparing the two methods. Serial dilutions of samples (10, 50, 100, or 500 mg of the cutting oil added to 300 ml of distilled water in a beaker) were analyzed by the TOC method. Similar samples were added to 150 ml of distilled water and placed in a Greenburg-Smith impinger; approximately 1.3 dry standard cubic meters (dscm) (45 dry standard cubic ft (dscf)) of air was bubbled through the impinger, and the sample was recovered and analyzed by ether-chloroform extraction. Table 4 summarizes the results of this analysis. Table 5 compares the analyses by the TOC and by ether-chloroform extraction methods when the cutting oil was added to a Method 5 train. Accuracy and reproducibility were higher for the ether-chloroform extraction method than for the TOC method. Average recovery was slightly greater than 100%. TOC analysis gave low recovery and highly variable results due, at least in part, to the technique of obtaining the

TABLE 4. DATA ON DILUTED CUTTING OIL[a]

Sample volume, μℓ	Sample weight, mg	Sample recovered	
		weight, mg	percent, %
10	9.3	8.7	93.5
50	46.0	49.6	107.8
100	89.2	93.7	105.0
500	447.8	450.6	100.6

[a]Cutting oil added to 300 ml of distilled water in a separatory funnel, then extracted by ether-chloroform.

TABLE 5. ETHER-CHLOROFORM AND TOC ANALYSES OF DILUTED CUTTING OIL ADDED TO A METHOD 5 TRAIN

Sample volume μℓ	Sample weight mg	Weight recovered, mg			Proportion recovered, %	
		Extraction[a]	Acetone rinse	TOC	Ether-Chloroform extraction[a]	TOC[b]
10	9.3	6.0	5.0	0.3	118.3	57.0
50	46.0	27.5	13.5	2.3	89.1	34.3
100	89.2	70.9	17.6	4.3	99.2	30.9
500	447.8	277.2	131.9	24.6	91.4	56.5

[a]Based on weight of organic fraction recovered by ether-chloroform extraction and by the acetone rinse of the impinger.

[b]Based on weight recovered by total organic carbon (TOC) analyzer and by the acetone rinse of the impinger.

aliquot for analysis, indicating that the variations between the analytical methods shown in Table 3 may be due to the variability of the TOC method and not the ether-chloroform extraction method. A solution of cutting oil and water may, in fact, be made homogeneous by using an inorganic emulsifier, but this is not recommended since the reproducibility of values obtained with ether-chloroform extraction was quite acceptable.

Hydrocarbon Collection Efficiency

The efficiency of the sampling train in collecting hydrocarbons was checked at Plant A by using a GC/FID to measure hydrocarbons in the gas leaving the third impinger and by comparing this value to that of the stack gas in the vicinity of the nozzle.

A heated Beckman model total hydrocarbon analyzer was used to measure the total hydrocarbon concentration exiting the stack. First, a sample was taken out of the stack through a heated line into the FID, which recorded the total hydrocarbon concentration. Next, a sample which had gone through the modified Method 5 sampling train was analyzed with the GC/FID. The sampling train removed about 98% of the material in the stack gas that gave a response to the GC/FID. There was no correlation between the total emissions in the sampling train residue and the emissions measured by the GC/FID.

Sulfate Content Analysis

The sulfate content of the filterable particulate catch was determined at Plant A. In 24 samples from the windbox vent, the sulfate concentration averaged 8.01 mg/sm^3 with an SD of 7.04 mg/sm^3. Values ranged from 1.65 to 27.43 mg/sm^3.

RESULTS OF PLANT B STUDY

Analysis of the windbox emissions from Plant B are shown in Table 6. Five of the sets were run at a reduced filter temperature. The filterable particulate emissions at Plant B were about twice as high as than those at Plant A and more variable. The average filterable particulate concentration was 48.4 mg/sm^3 with an SD of 21.34 mg/sm^3. Runs GA-2 and GB-2 were outliers and >5 SD's from the run mean, so these two runs were not included in the average SD.

The precision, measured by CV, of the filterable particulates data was estimated to be 44%. The run sets of four samples (Table 7) gave grouped data with an average CV of approximately 20%. The mean value for all valid OSE catches was 18.28 mg/sm^3 with an SD of 15.17 mg/sm^3, and a CV of 83%. A similar estimate for the grouped data showed an average CV of about 30%. The large difference in CV's for filterable particulate (they range from 4 to 51) shows the effect of differences in the process conditions at different times. During the testing no efforts were made to hold the process constant; testing was done during all the times when the sinter line was on, even if it was operating at a very low level.

The large difference between the grouped and ungrouped data is probably due to the effect of changing process conditions.

Sampling Filtration Temperature Effects

To determine if the sampling filtration temperature was critical to the collection of the mass of filterable particulates, five of the seven run sets were operated with temperature

TABLE 6. RESULTS FROM PLANT B[a]

Test number	Sample volume DSCM	Filterable particulate CS mg/sm³	Impinger OSE mg/sm³	Total[b]	Total organic's CS mg/sm³	Total organic carbon CS mg/sm³	Impinger inorganic's CS mg/sm³	Impinger[d] sulfates CS mg/sm³
Filter temperature ≅ 120°C (250°F)								
GA-1	2.51	32.07	16.04	48.11	26.15	5.89	4.62	9.50
GB-1	2.45	30.34	34.59	64.93	52.76	9.23	12.90	11.46
GC-1	2.44	32.85	15.48	48.33	64.42	6.88	8.93	6.02
GD-1	2.41	32.90	17.65	50.55	47.65	4.60	8.00	7.38
GA-2	0.99	37.97	3366.53	3404.50	3404.00	18.28	1275.37	25.02
GB-2	1.01	24.87	2244.38	2269.25	2272.68	8.88	996.90	15.84
GA-3	2.42	97.95	1.86	99.81	11.35	6.48	82.76	9.60
GB-3	2.34	90.17	1.45	91.62	9.77	11.05	19.71	9.09
GC-3	2.39	90.81	2.05	92.86	6.74	4.10	183.12	6.98
GD-3	2.35	51.91	1.95	53.86	15.07	1540.86	63.86	7.97
GA-4	1.65	64.78	36.92	101.70	53.20	5.69	16.28	22.79
GB-4	1.59	43.18	39.22	82.40	55.37	9.55	16.22	16.88
GA-5	1.11	48.25	18.72	66.97	55.90	18.18	8.64	19.31
GB-5	1.07	48.05	26.03	74.08	51.50	20.34	13.25	17.21
GC-6	1.54	43.39	8.50	51.89	17.25	3.63	73.35	4.47
GD-6	1.45	22.72	11.36	34.08	24.58	6.61	21.41	3.83
GC-7	1.15	39.42	9.66	49.08	27.32	6.27	23.76	3.92
GD-7	1.13	29.80	4.86	34.66	18.22	9.20	19.19	3.45

(continued)

TABLE 6 (continued)

Test number	Sample volume DSCM	Filterable particulate CS mg/sm³	Impinger OSE mg/sm³	Total[b]	Total organic's CS mg/sm³	Total organic carbon CS mg/sm³	Impinger inorganic's CS mg/sm³	Impinger[d] sulfates CS mg/sm³
Filter temperature ≅ 65°C (150°F) to 93°C (200°F)								
GC-2[e]	1.02	42.63	369.49	412.12	383.78	8.33	191.99	13.53
GD-2	0.97	29.04	31.93	60.97	71.78	7.11	18.74	14.55
GC-4	1.63	40.00	50.83	90.83	65.33	6.79	28.50	16.88
GD-4	1.59	46.73	44.02	90.75	169.95	6.05	24.47	19.97
GC-5	1.07	51.85	28.35	80.20	50.27	8.02	12.68	20.97
GC-5	1.07	58.56	35.04	93.60	49.45	11.34	18.13	17.99
GA-6	1.54	50.74	0.33	51.07	19.67	3.07	32.14	3.60
GB-6	1.38	84.10	7.34	91.44	20.94	2.98	21.08	3.50
GA-7	1.19	47.92	5.05	52.97	21.61	3.53	19.25	3.92
GB-7	1.12	36.84	12.64	49.48	35.95	4.00	16.29	4.14

[a]Stack conditions - 50°C (120°F), 14% H_2O.

[b]Filterable particulate plus impinger OSE.

[c]Total chromatographable organics.

[d]Water soluble sulfates of the impinger contents.

[e]Runs GC-2 and GD-2 were run at 65°C (150°F) but moisture conditions necessitated raising the temperature to 93°C (200°F) for the rest of the reduced filter temperature runs.

as a variable. The data are shown in Table 6. Two of the four trains in a single set were sampled at the conventional 120°C (250°F) of Method 5; the other two were operated at a reduced temperature of approximately 65°-82°C (150°-180°F). The lower temperature could not be precisely controlled due to considerable amounts of condensed water in the gas. The samples from the reduced-temperature trains were expected to show higher filterable particulate catches than the standard-temperature trains due to the condensation of additional organics on the filter. The OSE catch was expected to be reduced due to the deposition of organics on the filter. Analysis of the paired train data showed that four of the five filterable particulate samples had increases in the front-half catch. Unfortunately, the OSE results did not fit the model; that is, only one set of OSE data showed the expected decrease. When the means of the paired data were compared statistically by using the Students t-test, none of the differences between the means were significant at the 90% confidence level.

Results of Special Laboratory Analysis

Organics were measured before extraction and evaporation using a procedure called total chromatographable organics (TCO). The TCO analysis indicated that the extraction-evaporation procedure would lose considerable amounts of the organics. As noted earlier it was for this reason that the term OSE is used in this report to emphasize that this catch depends on the means of recovery, and is not a measure or total organic emissions.

Data Precision

The CV calculations are shown in Table 7. For the filterable particulate data, the average CV is 19%--a value at least twice that found for some other well controlled sources using EPA Reference Method 5.[5]

Plant B had been tested twice before with the same method at times when the process conditions were stable. Table 8 shows the CV data for the previous tests; results of the current study agree with the results of the earlier work, even though the magnitudes of emissions were not the same. These data may suggest that the organic or sulfate components of the filterable particulate are more variable for sintering plants than for power plants or municipal incinerators yet adequate data to substantiate this statement is not included in this study and further analysis would be required before any definitive conclusions could be drawn.

Sample Recovery

Sample recovery from the impingers at Plant B consisted of recovering the impinger water, rinsing it with distilled water, then with acetone, and finally rinsing it with methylene chloride. The methylene chloride rinse removed approximately five times as much organic material as the acetone rinse. Most of the material removed by the acetone and methylene chloride was organic. The methylene chloride rinse was not done with the OSE since the chemical composition of this rinse was not known. Preliminary GC studies showed that it contained only one large peak, which was not identified.

TABLE 7. STATISTICAL RESULTS OF GROUPED RUN SET DATA - PLANT B

Run set	Temperature, °C (°F)	Mean ($\bar{x}$) FP[a] mg/sm³	Mean ($\bar{x}$) OSE[b] mg/sm³	Mean ($\bar{x}$) Total[c] mg/sm³	Standard deviation (SD) FP mg/sm³	Standard deviation (SD) OSE mg/sm³	Standard deviation (SD) Total mg/sm³	Coefficient of variation (CV = SD/$\bar{x}$ × 100) FP, %	CV OSE, %	CV Total
1/A-D	120 (250)	32.04	20.94	52.98	1.20	9.15	8.04	4	43	15
2[d]/A,B	120 (250)	31.42	-	-	9.26	-	-	29	-	-
C,D	65 (150)	35.84	-	-	9.61	-	-	27	-	-
A-D	-	33.63	-	-	8.12	-	-	24	-	-
3[e]/A-D	120 (250)	82.71	1.83	84.54	20.8	0.26	20.77	-	14	-
A-C	120 (250)	92.98	1.79	94.76	4.32	0.31	4.41	5	-	5
4/A,B	120 (250)	53.98	38.07	92.05	15.27	1.63	13.65	28	-	-
C,D	65 (150)	43.73	47.43	90.79	4.76	4.82	0.06	10	-	-
A-D	-	48.67	42.74	91.42	11.10	6.14	7.91	22	14	9
5/A,B	120 (250)	48.15	22.38	70.53	0.14	5.17	5.03	0.3	-	-
C,D	65 (150)	55.21	31.70	86.90	4.74	4.73	9.48	9.	-	-
A-D	-	51.68	27.04	78.71	4.91	6.73	11.30	8.	25	14
6[f]/C,D	120 (250)	33.06	9.93	42.99	14.62	2.02	12.59	44	-	-
A,B	65 (150)	67.42	3.84	71.26	23.59	4.96	28.55	35	-	-
A-D	-	50.24	6.88	57.12	25.74*	4.68	24.31	51	-	-
B-D	-	50.07	9.07	59.14	31.23	2.07	29.36	-	23	50
7/C,D	120 (250)	34.61	7.26	41.87	6.80	3.39	10.20	20	-	-
A,B	65 (150)	42.38	8.85	51.23	7.83	5.37	2.47	18	-	-
A-D	-	38.50	8.05	46.55	7.48	3.78	8.12	19	47	17

[a]Filterable particulate.

[b]Organic solvent extractables.

[c]FP plus OSE.

[d]Outlier values for organics.

[e]FP acetone rinse for Run D presumed invalid.

[f]OSE for Run A presumed invalid.

TABLE 8. PRECISION OF PLANT B TEST DATA

Study	Coefficient of variaton (CV)	
	Filterable particulate, %	OSE, %
1975	18	31
1976	25	41
1978	19	28

Sulfate Content

The sulfate contents of the impinger catch were analyzed to determine the effects of filter temperature on the formation of sulfates. It is likely that much of the inorganic material in the back-half catch resulted from the formation of sulfates in the cooled impinger water, but further study is needed for verification.

5.0 REFERENCES

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3. ASTM Standard Method D2778-70, reapproved in 1974.

4. McGaughey, J. F., and D. E. Wagoner. Special Analyses of Samples from Sinter Plants in the Iron and Steel Industry. EPA Contract No. 68-02-2725, RTI/1487/64-01F, January 1978.

5. Mitchell, W. J., and M. R. Midgett. Means to Evaluate Performance of Stationary Source Test Methods. E.S.&T 10, 85-88 (1976).

Note: Computer printouts, field data, laboratory results, sampling and analytical procedures, and calibration procedures and Results have been printed in limited quantities in the form of additional volumes to this report and are available upon request.

APPENDIX: ANALYTICAL PROTOCOL

1.0 PLANT A

1.1 Front Half

1. Heat the filters and the probe washes for Tests 5 and 8 at 200°C (390°F) for 4 h, and reweigh to determine weight loss.

2. Combine the filters from Samples C and D of Tests 5 and 8 with the appropriate probe washes, extract directly with water by using an ultrasonic bath, and determine the sulfates ($SO_4^=$) in the extract.

3. Combine the filters from Samples A and B of Tests 5 and 8 with the appropriate probe washes, and extract the organic material by using a 1:1 mixture of ether-chloroform solvent; backwash the solvent with water, and add the water wash to the extracted filter and probe wash in an ultrasonic bath; determine the $SO_4^=$ in the water extract by titrating with barium perchlorate and determine the organic content by evaporating the ether-chloroform extract.

4. Use the following evaluation criteria:

a. In step 1, if the loss of material was $\leq$5% at 200°C (390°F), proceed with Step 3, but if the loss was >5%, hold and discuss with the EPA project officer.

b. In setps 2 and 3, if the $SO_4^=$ ion content has not changed, proceed with the following test after determining the $SO_4^=$ in the remaining samples (Step 3). If the $SO_4^=$ has changed, there is no need for $SO_4^=$ analysis of the rest of the samples.

1.2 Water Catches

1. Determine the total organic carbon (TOC) content from all impinger catches.

2. Measure the volumes, and then sequentially extract the impinger water at pH's 3.0, 11.0, and 7.0. (Use standardized HCl and NaOH to adjust the pH's.) Record all volumes of standardized reagents.

3. Do all extractions at an aqueous-organic phase ratio of 10.

4. Determine the residue from all extractions and from the aqueous raffinate.

5. Use the following evaluation criteria:

a. If the extracted residues at pH's 11 and 7 are relatively insignificant compared to that at pH 3, discontinue the extractions; that is, extract the remaining samples at pH 3 only.

6. Correct the residues from the aqueous raffinate for the Cl^- or Na^+ ions added during the HCL and NAOH adjustments of pH.

1.3 Back Washes of Impingers (Acetone)

1. Measure the volumes, and then evaporate the acetone to dryness at room temperature.

2. Determine the residue gravimetrically.

1.4 Backup Filter (Between Third and Fourth Impingers)

1. Place the backup filter in acetone, and then filter off the acetone; continue to wash the filter repeatedly with spectro-quality acetone.

2. Evaporate the filtered acetone at room temperature, and measure the residue gravimetrically.

2.0 Plant B

2.1 Front Half

1. Dry the filter on a watchglass in a forced-air oven at 100° to 105°C (210° to 220°F) for 24 h, cool in a desiccator over Drierite (anhydrous $CaSO_4$), and weigh to the nearest 0.1 mg; place in the desiccator for 6 h and reweigh. If the difference between the two weights is >0.5 mg, either redry the filter at intervals of at least 6 h until two consecutive weights are within 0.5 mg or for 24 h before the first reweighing.

2. Average the two gross weights determined above, subtract the tare, and record the net weight in milligrams.

3. Return the filter in its petri dish; reseal and store.

2.2 Probe and Impinger Washes (Acetone)

1. Measure and record the volume of the sample to the nearest 10 ml.

2. Label a 150-ml beaker with the sample number and "acetone;" dry the beaker in an oven at 105°C (220°F) for 2 h; cool the beaker in a desiccator over Drierite; and tare-weigh it to the nearest 0.1 mg.

3. Pour the sample into the weighed beaker, and rinse the beaker with a minimum amount of acetone.

4. Set the beaker under a fume hood until the acetone evaporates; if any water remains, oven dry the beaker at 105°C (220°F), and put it in a desiccator over Drierite for at least 1 h after air or oven drying.

5. Weigh the beaker to the nearest 0.1 mg at intervals of at least 6 h until two weights are within $\leq$0.5 mg of each other.

6. Average the two gross weights (Step 5) subtract from it the tare weight (Step 2), and record the net weight in milligrams. If the net weight is <10 mg, cover the beaker with Parafilm,* and store it; if it is $\geq$10 mg, proceed to the next steps.

7. Pipette 50 ml of deionized water into the beaker, and scrape the walls and bottom with a plastic spatula or rubber policeman to dissolve as much material as possible.

8. Pipette 20 ml of the sample from the beaker into a clean, dry 50-ml plastic bottle, but without rinsing the pipette with the sample before the transfer; then set the bottle aside for later analyses.

9. Pour the remaining 30 ml of the sample from the beaker into a 60-ml separatory funnel, rinse the beaker with a few

*Registered trademark.

milliliters of water and add the rinse to the separatory funnel; pour 5 ml of diethyl ether into the separatory funnel, stopper it, shake it for about 1 min, release the pressure with the stopcock, allow the layers to separate, and drain the bottom (i.e., the aqueous phase) into the beaker.

10. Tare two 150-ml beakers (Step 2) label one "organic" and the other "aqueous," and label both with the sample number.

11. Drain the ether from the separatory funnel into the "organic" beaker, and then pour the water from the beaker back into the funnel; repeat the ether extraction twice (Step 9), draining the ether layer into the "organic" beaker each time.

12. Pour the water back into the separatory funnel; pour 5 ml of chloroform into the beaker, rinse gently, and pour it into the separatory funnel; stopper and shake for 1 min, and release the pressure with the stopcock, allow the layers to separate, and drain the lower layer ($CHCL_3$) into the "organic" beaker.

13. Repeat Step 12 twice (except for putting chloroform directly into the separatory funnel); drain both additional aliquots into the "organic" beaker, and put the beaker under a fume hood until the organic solvents evaporate; and then follow the weighing procedure (Step 5).

14. Drain the water from the separatory funnel into the "aqueous" beaker. Put this beaker into a drying oven at 105°C until dry; cool it in a desiccator; and follow the same weighing procedure as described in Steps 4 and 5.

15. Calculate the gross and net weights of the organic and inorganic fractions (Step 6).

16. Follow Steps 1-16 above, using 100 ml of acetone as a blank.

2.3 Impinger Wash (Methylene Chloride)

1. Follow Steps 1-7 for the acetone washes, but label the beaker (Step 2) with the sample number and "M.C." for methylene chloride (instead of "acetone").

2. Pipette 30 ml of deionized water into the beaker and scrape the walls and bottom with a plastic spatula or rubber policeman to dissolve as much material as possible; measure the pH of the solution to within $\pm$0.02 units.

3. Pour the sample into a 60-ml separatory funnel, rinse the beaker with a few milliliters of water, and add the rinse to the separatory funnel; pour 5 ml of methylene chloride into the beaker, rinse it gently, pour it into the funnel, stopper it, shake it for 1 min, release the pressure with the stopcock, and allow the layers to separate.

4. Tare two 150-ml beakers; (Step 2, Section 2.2) label one "organic" and the other "aqueous" and label both with the sample number.

5. Drain the lower (i.e., the organic) layer from the separatory funnel into the "organic" beaker; repeat the extraction (Step 3) with two more 5-ml portions of methylene chloride, putting both into the "organic" beaker; put the beaker under a fume hood until the organic solvents evaporate, and follow the weighing procedure (Steps 4 and 5).

6. Drain the water from the separatory funnel into the "aqueous" beaker; put the beaker into the oven at 105°C (220°F) until dry and cool it in a desiccator; follow the procedure for calculating the gross and net weights of the organic and inorganic fractions (Steps 5 and 6, Section 2.2).

7. Follow the Steps 1-7 above, using 100 ml of methylene chloride as a blank.

2.4 Impinger Contents and Rinse (Water)

1. Combine the two water samples in the impinger contents container, and use the impinger content sample number thereafter; measure the pH of each sample within $\pm$0.02 units.

2. Pour an aliquot of the combined sample for TOC analysis in a 50-ml plastic bottle, and acidify it to a pH of <2 with concentrated H_2SO_4.

3. Titrate a 20-ml aliquot of the combined sample for $SO_4^=$, following the procedure in Section 5.5.6.

4. Rinse a 1-ℓ beaker with chloroform and allow it to air dry; measure out 500 ml of water with a graduated cylinder, use 300 ml if necessary, but record the volume used; pour it, and then set up a pH electrode in it.

5. Titrate the water to a pH of 7.0 with 0.5N NaOH (20 g dissolved in H_2O and diluted to 1ℓ), and record the volume of titrant used.

6. Prepare one tared 150-ml beaker and one tared 250-ml beaker (Step 2, Section 2.2); label the 150-ml beaker "organic" and the 250-ml beaker "aqueous," and label both with the sample number.

7. Pour the water from the 1-ℓ beaker into a 1-ℓ separatory funnel, add 25 ml of diethyl ether, stopper it, shake it for about 1 min, release the pressure with the stopcock, allow the layers to separate, and drain the bottom (i.e., the aqueous layer) back into the 1-ℓ beaker.

8. Drain the ether from the separatory funnel into the "organic" beaker, and pour the water back into the funnel; repeat the ether extraction twice (Step 7), draining the ether layer into the "organic" beaker each time.

9. Pour the water back into the separatory funnel; rinse the 1-ℓ beaker with 25 ml of chloroform, pour the rinse into the separatory funnel, stopper it, shake it for 1 min, release the pressure with the stopcock, allow the layers to separate, and drain the lower (i.e., the organic) layer into the "organic" beaker.

10. Repeat Step 9 twice, but put the chloroform directly into the separatory funnel; drain both additional chloroform layers into the "organic" beaker; put the beaker under a fume hood until the organic solvents evaporate; and follow the weighing procedure (Steps 4 and 5, Section 2.2).

11. Drain as much of the water as possible from the separatory funnel into the "aqueous" beaker; put the beaker on a hot plate, evaporate most of the water just below the boiling point, pour the remaining water from the funnel into the 1-ℓ beaker, and cover it; when the "aqueous" beaker has room, pour the rest of the water into it, rinse the 1-ℓ beaker with the water, and add the rinse to the "aqueous" beaker.

12. Finish evaporating the water in a drying oven at 105°C (220°F); cool in a desiccator; and follow the weighing procedure (Steps 4 and 5, Section 2.2) to calculate the gross and net weights of the organic and inorganic fractions.

13. Follow Steps 1-12 above with 500 ml of deionized water.

14. Calculate the total amount of organic and inorganic material in the sample:

$$\text{Total} = \frac{\text{net weight} \times \text{initial volume}}{\text{volume extracted}}.$$

2.5 Backup Filter (Acetone)

1. Keep the filter and the acetone in their original containers, and place them in an ultrasonic bath for 30 min.

2. Label, oven dry, desiccate, and tare-weigh a 150-ml beaker.

3. Rinse a vacuum filtration apparatus (Buchner funnel and sidearm flask) with acetone and allow it to dry.

4. Vacuum filter the sample through a Whatman GF/A filter or the equivalent; allow the back-half filter to rest in the Buchner funnel, and rinse the bottle and both filters with acetone.

5. Pour the filtrate into the beaker, rinse the sidearm flask with acetone, and add the rinses to the beaker.

6. Put the beaker under a fume hood until the acetone evaporates, and then follow the weighing procedure (Steps 4 and 5, Section 2.2).

7. Discard the two filters.